ELSEVIER

Journal of Magnetism and Magnetic Materials 202 (1999) 480–484

Journal of magnetism and magnetic materials

www.elsevier.com/locate/jmmm

# Exchange biasing in ferromagnet/antiferromagnet Fe/$KMnF_3$

Z. Celinski[a,*], D. Lucic[a], N. Cramer[a], R.E. Camley[a], R.B. Goldfarb[b], D. Skrzypek[c]

[a]*Department of Physics, University of Colorado, Colorado Springs, CO 80918, USA*
[b]*Electromagnetic Technology Division, National Institute of Standards and Technology, Boulder, CO 80303, USA*
[c]*Institute of Physics, University of Silesia, Uniwersytecka 4, Katowice, 40-007, Poland*

Received 18 September 1998; received in revised form 15 February 1999

**Abstract**

A new ferromagnet/antiferromagnet bilayer system, Fe/$KMnF_3$, exhibits interesting interfacial exchange properties. The bulk antiferromagnet $KMnF_3$ has three possible magnetic states: paramagnetic, antiferromagnetic, and weakly ferromagnetic spin-canted. Consequently, the exchange anisotropy in Fe/$KMnF_3$ is unusual. We examine the exchange bias in Fe/$KMnF_3$ as a function of the magnetic state. Monocrystalline Fe(0 0 1) and polycrystalline Fe films, 3 nm thick, were grown epitaxially on Ag(0 0 1) templates on GaAs(0 0 1) substrates. Epitaxial $KMnF_3$ was then grown on both the single-crystal and polycrystal Fe. We measured the low-field, zero-field-cooled and field-cooled magnetizations as functions of temperature. The zero-field-cooled single-crystal Fe magnetization is greatly reduced at liquid-helium temperatures. We see the influence of the transition from the antiferromagnetic to the spin-canted state on the exchange coupling. The blocking temperature is close to the Néel temperature (89 K). From the shift in the hysteresis loop, we estimate the strength of the interfacial exchange coupling to be $4.5 \times 10^{-5}$ J/m$^2$. 

*PACS:* 75.30.Gw; 75.70.Cn

*Keywords:* Exchange biasing

## 1. Introduction

The interfacial exchange interaction between a ferromagnet and an antiferromagnet is an important, though not well understood, problem in fundamental magnetism, despite nearly 40 years of research since the discovery of exchange anisotropy by Meiklejohn and Bean [1] in the Co/CoO system. Interfacial exchange has technological applications in domain stabilization in magnetoresistive heads [2] and spin-valve-based devices [3].

Many factors seem to contribute to the strength of the exchange bias. These include intrinsic factors, such as the exchange stiffness and anisotropy in the antiferromagnet, as well as extrinsic factors, such as grain size, orientation, and texture. In addition, recent experiments and theoretical work have emphasized interfacial roughness as a critical parameter. For example, some work suggests that biasing is a result of an uncompensated surface of

* Corresponding author. Tel.: +1-719-2623283; fax: +1-719-2623013.
*E-mail address:* zcelinsk@alf.uccs.edu (Z. Celinski)

0304-8853/99/$ - see front matter 
PII: S0304-8853(99)00364-9

the antiferromagnet [4]. A recent theoretical model, in contrast, suggests that exchange biasing can occur at a magnetically compensated surface and may be the result of the creation of domain walls in the antiferromagnet [5]. The details and strength of this interaction depend on the orientation of the easy axis of the antiferromagnet with respect to the direction of the applied field [6]. To check the applicability of this theory and to determine the importance of interfacial roughness and crystallographic structure, it is necessary to have a structurally well defined, single-crystal system. Here, we investigate the interaction between the ferromagnet and antiferromagnet in single-crystal and polycrystal Fe/$KMnF_3$ structures.

The fluoride $KMnF_3$ exhibits unusual magnetic and structural properties [7–9]. We examine the exchange coupling between an Fe film and $KMnF_3$ as a function of the fluoride's magnetic structure. Between room and liquid-nitrogen temperatures, the bulk material has three magnetic states: paramagnetic (above 89 K), antiferromagnetic (81–89 K), and the weakly ferromagnetic spin-canted state (below 81 K) [7–9]. In addition, the magnetic state of the fluoride can be changed from antiferromagnetic to spin-canted by applying an external magnetic field smaller than 1 T. In the antiferromagnetic state in a bulk crystal, the magnetic moments of the Mn atoms are along the $z$-axis. In contrast, in the spin-canted state there is a spin reorientation and the moments are perpendicular to the $z$-axis [8,10]. The canting itself is less than one degree. Of course, the spin configuration in a thin film could be different, but it is likely that a substantial spin reorientation takes place with the transition from an antiferromagnetic state to the spin canted state. The ability to change the magnetic structure of the antiferromagnet allows us to experimentally probe how exchange coupling depends on magnetic structure.

$KMnF_3$ is a perovskite cubic crystal [11] with a lattice constant of 0.419 nm. An important question concerns the nature of the interface between $KMnF_3$ and Fe. Fig. 1 shows the (0 0 1) surface of $KMnF_3$, and how the Fe atoms would fit into the resulting four-fold hollow surface. The lattice mismatch between the expected Fe positions and bulk Fe lattice spacing is about 3%. Thus we can expect that epitaxial growth of Fe/$KMnF_3$ using molecular beam epitaxy (MBE) could create ferromagnet/antiferromagnet structures with well defined interfaces.

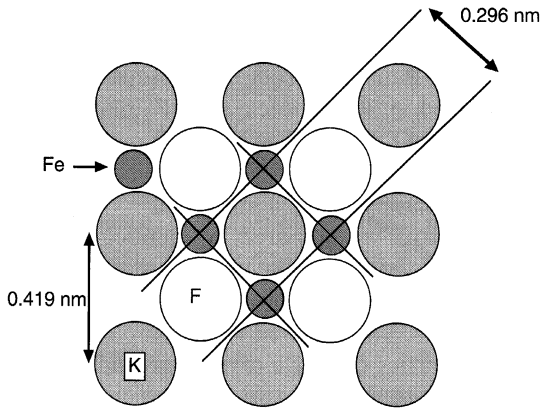


Fig. 1. Illustration of the surface geometry of the (0 0 1) surface of $KMnF_3$ and how the Fe atoms fit into this surface. The atoms are drawn to scale with the atomic radii.

## 2. Experimental details

Fe/$KMnF_3$ bilayers were grown in an ultrahigh vacuum (UHV) MBE system at $2 \times 10^{-8}$ Pa. Fe and Ag were evaporated from K-cells and $KMnF_3$ was evaporated using an electron gun. Growth temperature was about 325 K. No field was applied during deposition. We used GaAs(0 0 1) substrates prepared by a sequence of annealing and sputtering at 820 K. We observed the characteristic reconstruction in the reflection high energy electron diffraction (RHEED) pattern for GaAs, indicating a gallium-terminated surface. On this surface we grew a thin (1 nm) seed layer of Fe followed by a Ag film (100 nm). During growth we observed that both the Fe seed layer and the Ag template were single crystal. However, to improve the Ag template quality, we annealed it at 620 K for about 10 h in UHV.

An Fe film, 20 monolayers thick, was then grown on the Ag template. Its structural quality was monitored by observation of RHEED intensity oscillations, which indicated pseudo-layer-by-layer growth, and by the RHEED patterns themselves.

From the RHEED results we estimate that the Fe terraces have a horizontal width on the order of 25 nm. The four-fold structural symmetry was well pronounced. A 30 nm thick $KMnF_3$ film was then grown on the Fe layer. The RHEED pattern clearly indicated a single-crystal $KMnF_3$ film. The structures were capped with 4 nm of Au.

In addition, we prepared an identical structure with all polycrystalline layers. These films had no Fe seed layer and the polycrystalline Ag layer was not annealed. This allowed us to compare the magnetic properties of single-crystalline and polycrystalline $Fe/KMnF_3$ structures.

We used a magnetometer based on a superconducting quantum interference device (SQUID) to study the magnetic properties of the bilayers. All samples were demagnetized at room temperature before SQUID measurement. We applied a field of 0.5 T during field cooling. The hysteresis loops of field-cooled samples were measured at 5 K. The single-crystal samples were measured along the easy (1 0 0) and hard (1 1 0) axes of the Fe film. The diamagnetic contribution of the substrate was subtracted from the magnetic data.

## 3. Results and discussion

Field cooling in 0.5 T to 5 K caused a negative shift in the hysteresis loops of both the single-crystal and polycrystal specimens, typical for exchange-coupled systems. The loop for the single-crystal structure shifted 8.5 mT, twice as much as for the polycrystal sample, 3.5 mT. Such shifts, using the simplest model, indicate an exchange interaction between the ferromagnet and the antiferromagnet on the order of $4.5 \times 10^{-5}$ J/m$^2$ for the single crystal and $2 \times 10^{-5}$ J/m$^2$ for the polycrystal.

Single-crystal samples allow us to probe the connection between the magnetic response and crystallographic orientation. In uncoupled Fe films, hysteresis loops along the easy and hard in-plane axes are generally quite different; the easy-axis loop is rectangular whereas the hard-axis loop is slanted and much narrower. In the $Fe/KMnF_3$ bilayer structure at 5 K, both the easy- and hard-axis hysteresis loops are rectangular and similar in width,

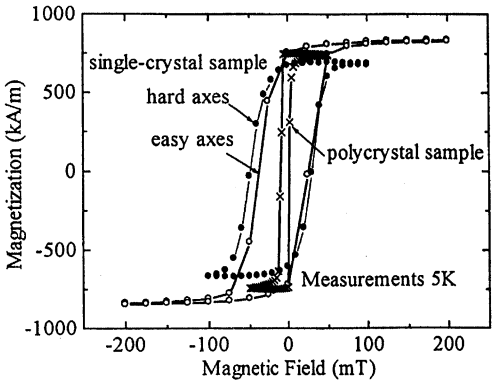


Fig. 2. Hysteresis curves for $Fe/KMnF_3$ at 5 K. The samples were cooled in a field of 500 mT. The coercivity is substantially larger for the single-crystal sample.

about 35 mT (Fig. 2). In contrast, the polycrystal sample, with no unique crystallographic orientation, has a much narrower loop, about 6 mT. This shows the importance of the crystallographic structure of the $KMnF_3$ on the magnetic properties of the bilayer.

It is not immediately obvious if the magnetization of the $KMnF_3$ contributes to the magnetization measured in our experiment. From previous work [7], however, it is estimated that the spin canting of the $KMnF_3$ is less than one degree, even at low temperatures. Thus the $KMnF_3$ is very nearly in the antiferromagnetic state and its net magnetization is negligible compared to that from the Fe film.

Zero-field cooled magnetization at 5 K for the single-crystal sample along an easy axis of the Fe film required over 110 mT to saturate (Fig. 3), a value much larger than would be expected for an uncoupled Fe film. This indicates that the single-crystal $KMnF_3$ locks in the moments of the demagnetized Fe upon cooling, although the spin-canted structure of the $KMnF_3$ could cause Fe alignment out of the field direction.

A comparison of field-cooled (FC) and zero-field-cooled (ZFC) magnetization at 5 mT as functions of increasing temperature demonstrate the usual bifurcation for exchange-coupled layers. The FC magnetizations upon increasing temperature are similar (Figs. 4 and 5). The ZFC curves,

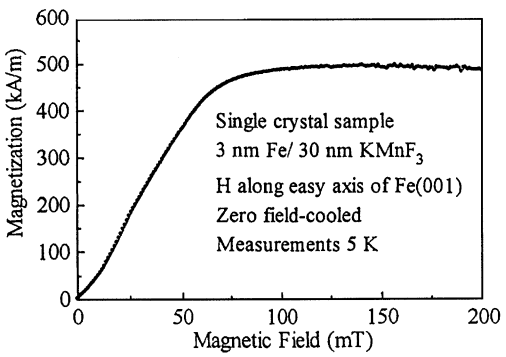


Fig. 3. Zero-field-cooled magnetization as a function of applied field at 5 K for the single-crystal sample. The saturation field is above 100 mT.

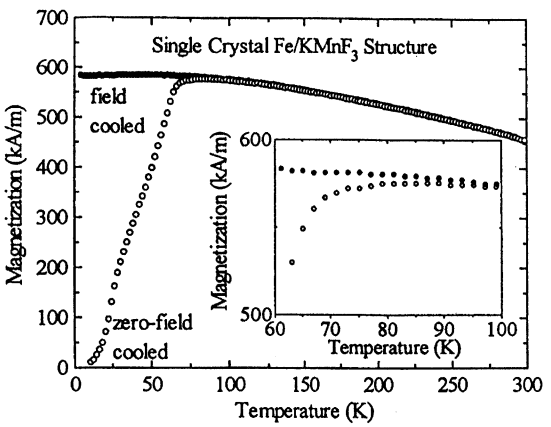


Fig. 4. Magnetization at 5 mT versus temperature for single-crystal Fe (3 nm)/$KMnF_3$ (30 nm) along an Fe easy axis. The FC sample was originally cooled in 500 mT. The ZFC magnetization is greatly reduced at low temperature. The inset shows the magnetization of $KMnF_3$ near the transition from the spin-canted to the antiferromagnetic state (after Refs. [6–8]).

however, are different. For single-crystal Fe/$KMnF_3$, the magnetization of the Fe film is close to zero at liquid He temperatures, indicating that the demagnetized Fe magnetization is frozen by the single-crystal fluoride at low temperatures (Fig. 4). The ZFC magnetization approaches the FC magnetization at 70 K and remains constant over the next 20 K. Both ZFC and FC magnetizations merge at the blocking temperature, 90 K. In contrast, ZFC and FC curves are similar to each other for the polycrystal sample, also merging at 90 K (Fig. 5).

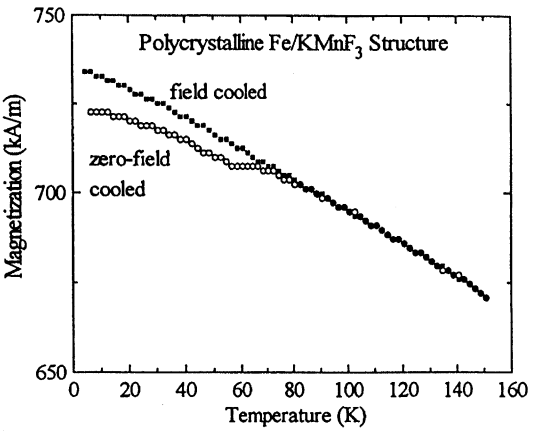


Fig. 5. Magnetization at 5 mT versus temperature for polycrystal Fe (3.5 nm)/$KMnF_3$ (30 nm) field. The FC sample was originally cooled in 500 mT. The ZFC and FC magnetizations are similar, in contrast to Fig. 4.

As discussed above, $KMnF_3$ has three magnetic states and provides an interesting opportunity to see how the magnetic state influences the exchange coupling. Bulk $KMnF_3$ is in the spin-canted state from 0 to about 81 K; the antiferromagnetic state then extends to about 89 K. The different regions of the ZFC magnetization curve for the single-crystal sample correspond to these known magnetic transitions in bulk $KMnF_3$. These critical temperatures are close to temperatures (Fig. 4) where the magnetization undergoes significant changes. The 90 K blocking temperature is close to the 89 K Néel temperature of $KMnF_3$.

## Acknowledgements

Steve Russek, NIST, provided helpful comments on the manuscript. The work at UCCS was supported by the Research Corporation and by the US Army Research Office through grants CC4084, DAAG55-97-1-0232, and DAAG55-98-0294. DS was supported by the State Committee for Scientific Research (KBN) in Poland through a statutory fund of University of Silesia.

## References

[1] W.H. Meiklejohn, C.P. Bean, Phys. Rev. 102 (1957) 1413.
[2] C. Tsang, R. Fontana, IEEE Trans. Magn. 18 (1982) 1149.
[3] B. Dieny, V.S. Speriosu, S.S.P. Parkin, J.C. Scott, B.A. Gurney, D.R. Wilhoit, D. Mauri, Phys. Rev. B 43 (1991) 1297.
[4] K. Takano, R.H. Kodama, A.E. Berkowitz, W. Cao, G. Thomas, J. Appl. Phys. 83 (1998) 6888.
[5] N.C. Koon, Phys. Rev. Lett. 78 (1997) 4865.
[6] R.E. Camley, R.J. Astalos, J. Magn. Magn. Mater. 198–199 (1999) 402.
[7] A.J. Heeger, O. Beckman, A.M. Portis, Phys. Rev. 123 (1961) 1652.
[8] M. Hidaka, J. Phys. Soc. Japan 39 (1975) 103.
[9] Z. Celinski, D. Skrzypek, Acta Phys. Polon. A 65 (1984) 149.
[10] M. Hidaka, N. Ohama, A. Okazaki, H. Sakashita, S. Yamakawa, Solid State Commun. 16 (1975) 1121.
[11] A. Ratuszna, D. Skrzypek, J. Kapusta, Phase Transitions 42 (1993) 189.